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CARBIDE COATED FIBERS IN GRAPHITE-ALUMINUM COMPOSITES

Progress Report No. 3: August 1 - December 31, 1974

Richard J. Imprescia, Leonard S. Levinson, Robert D. Reiswig, Terry C. Wallace, and Joel M. Williams

Prepared by
LOS ALAMOS SCIENTIFIC LABORATORY
Los Alamos, N. Mex. 87544
for Langley Research Center

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16. Abstract

This report describes the third part of an NASA-supported program at the Los Alamos Scientific Laboratory to develop graphite fiber-aluminum matrix composites. Thin, uniform coats of titanium carbide, deposited on graphite fibers by chemical vapor deposition with thicknesses up to approximately 0.1 μm were shown to improve fiber strength significantly. For greater thicknesses, strength was degraded. In addition, the coats promote wetting of the fibers and infiltration of the fiber yarns with aluminum alloys, and act as protective barriers to inhibit reaction between the fibers and the alloys.

Chemical vapor deposition was used to produce silicon carbide coats on graphite fibers. In general, the coats were nonuniform and were characterized by numerous surface irregularities. Despite these irregularities, infiltration of these fibers with aluminum alloys was good.

Small graphite-aluminum composite samples were produced by vacuum hot-pressing of aluminum-infiltrated graphite yarn at temperatures above the metal liquidus.

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SUMMARY

Thin, uniform coats of titanium carbide, deposited on graphite fibers by chemical vapor deposition with thicknesses up to approximately 0.1 μm, were shown to improve fiber strength significantly. For greater thicknesses, strength was degraded. In addition, the coats promote wetting of the fibers and infiltration of the fiber yarns with aluminum alloys, and act as protective barriers to inhibit reaction between the fibers and the alloys.

Chemical vapor deposition was used to produce silicon carbide coats on graphite fibers. In general, the coats were nonuniform and were characterized by numerous surface irregularities. Despite these irregularities, infiltration of these fibers with aluminum alloys was good.

Small graphite-aluminum composite samples were produced by vacuum hot-pressing of aluminum-infiltrated graphite yarn at temperatures above the metal liquidus.

INTRODUCTION

The work described here is the continuation of a NASA-supported program at the Los Alamos Scientific Laboratory (LASL) to develop graphite fiber-aluminum matrix composites. The LASL approach to this problem has been to use protective-coupling layers of refractory metal carbides on the graphite fibers prior to their incorporation into the composites. Such layers are directly wettable by liquid aluminum and should act as diffusion barriers to inhibit reaction between the graphite fibers and the aluminum.[1] Before pursuing the actual fabrication of a composite, however, the following preliminary objectives had to be achieved:

- development of a coating technique for producing thin, uniform coats of refractory metal carbides on the individual fibers of graphite yarns
- evaluation of the effect of carbide coats on the strength of the coated fibers
- demonstration of the ability of aluminum to wet carbide surfaces and infiltrate carbide-coated fiber yarns

- demonstration of the ability of carbide coats on graphite to inhibit reaction between graphite and aluminum.

Progress in these areas, reported previously, [1,2] is briefly reviewed here. Thin, smooth, continuous coats of zirconium carbide (ZrC) and titanium carbide (TiC) were uniformly deposited on the individual fibers throughout graphite yarns, using a batch chemical vapor deposition (CVD) process. Strength measurements on the TiC-coated fibers showed that strength can be influenced significantly by the presence of TiC coats on graphite fibers, but the relationship between coat thickness and strength was not established. Wetting of ZrC- and TiC-coated bulk graphite with aluminum was good, and moderate success was achieved in infiltrating carbide-coated fiber yarns. Initial experiments indicated that carbide coats on bulk graphite surfaces can act as effective diffusion barriers to inhibit reaction between graphite and aluminum. With these results, the feasibility of producing a graphite-aluminum composite, using carbide-coated graphite fibers, was established.

This report describes the results of the continuation of the above work plus activity in two additional areas. The specific objectives of the present work are:

- to evaluate the effect of coat thickness on the strength of carbide-coated graphite fibers
- to infiltrate carbide-coated graphite fiber yarns to a degree equivalent to that of commercially-infiltrated fiber yarns
- to evaluate the ability of carbide coats on graphite fibers to inhibit reaction between the fibers and an aluminum matrix
- to develop a batch CVD coating process for producing SiC coats on graphite fibers, and evaluate their influence on Al-infiltration, and their effectiveness as diffusion barriers in graphite-aluminum composites
- to develop a process for consolidating Al-infiltrated, carbide-coated fibers into small composite bars suitable for the measurement of mechanical properties.

Significant progress has been made in all these areas.

EXPERIMENTAL

Materials

Thornel 50, two-ply graphite yarn, obtained from Union Carbide Corporation, was used throughout the work described here. For the CVD experiments, three commercial grade, purified coating materials were used: titanium tetrachloride ($TiCl_4$), silicon tetrachloride ($SiCl_4$) and methyltrichlorosilane (CH_3SiCl_3). All of the infiltrations of graphite fiber yarns were done with LASL-produced Al-13 wt% Si alloys. Following is a typical analysis of one of these alloys:

TABLE I

COAT THICKNESS AND STRENGTH FOR
TiC-COATED THORNEL 50 GRAPHITE FIBERS

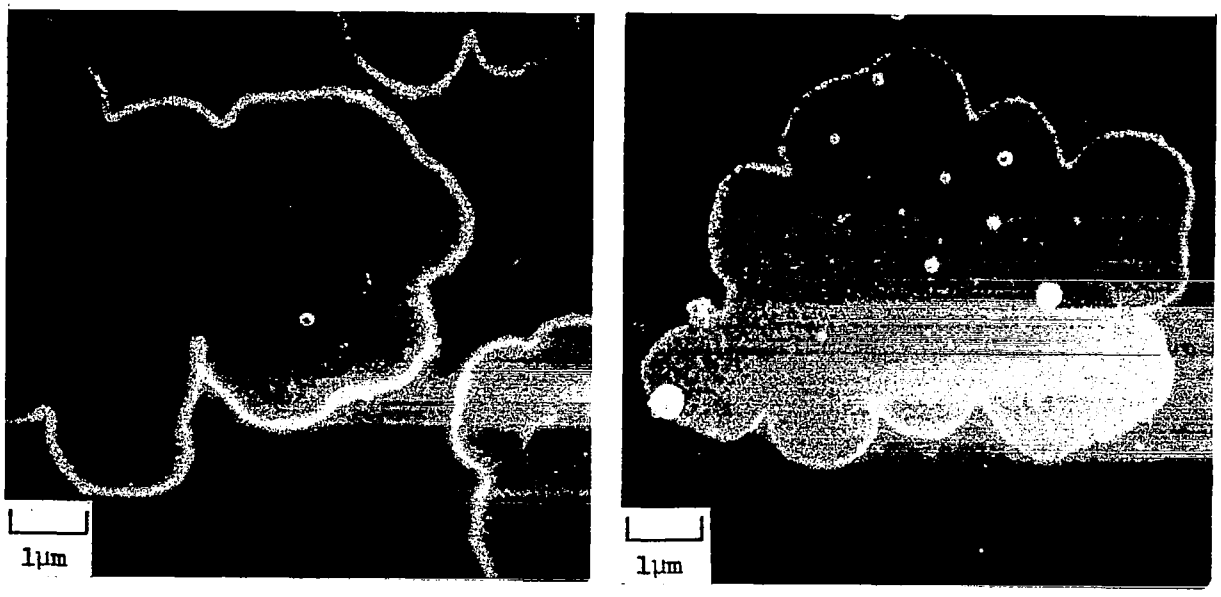
Run No.	Coat Thick., μm		Tensile Breaking Load, N (g-force)	
	Mean	Std. Dev.	Mean	Std. Dev.
5- 1-74	0.302	0.119	1.17 (119)	0.39 (40)
5- 2-74	0.069	0.040	14.78 (1507)	1.74 (177)
5- 3-74	0.105	0.045	10.78 (1099)	1.42 (144)
5-22-74	0.151	0.089	2.90 (296)	1.03 (105)
6- 4-74	0.078	0.028	11.93 (1217)	2.29 (234)
6- 5-74	0.125	0.053	7.57 (772)	1.60 (163)
6- 6-74	0.114	0.031	11.80 (1203)	1.28 (130)
6-19-74	0.122	0.049	4.43 (452)	1.01 (103)
6-24-74	0.148	0.093	9.37 (955)	1.06 (108)
6-25-74	0.123	0.037	11.71 (1194)	1.38 (141)
6-26-74	0.062	0.035	15.84 (1579)	1.90 (194)
6-27-74	0.104	0.054	14.55 (1484)	2.26 (230)
8-14-74	0.098	0.017	14.06 (1435)	1.63 (160)
Control[a]	0	---	11.36 (1158)	1.78 (182)

[a]Average of 135 tests on samples processed between 1023 K (750°C) and 1673 K (1400°C) without coating constituents in the gases.

12.7% Si, 0.4% Fe, 0.06% Cu, 0.04% Mg, 0.03% Mn, <0.01% Zn, remainder Al. From batch to batch the Si varied from about 11 to 13%.

Carbide Coated Fibers

TiC Coats.-Previously, it was demonstrated that thin, adherent coats of TiC could be uniformly deposited by CVD on the individual fibers of graphite yarns, using $TiCl_4$ as the coating gas.[2] It was also shown that these coats can significantly influence fiber strength. The relationship between strength and coat thickness, however, was not determined at that time. To evaluate this relationship, coat thicknesses were measured on 13 samples of TiC-coated Thornel 50 yarn and compared with the tensile breaking loads of the coated fibers. A tabulation of these data is given in Table I. The thicknesses were measured on photomicrographs, made through a scanning electron microscope (SEM), of cross sections of epoxy-mounted samples. Figure 1 shows two typical



(a) (b)

Fig. 1. Typical thin TiC coats on Thornel 50 fibers examined by SEM on polished cross-sections of epoxy-mounted samples. (a) Run 5-22-74, (b) Run 6-4-74.

examples of SEM photomicrographs prepared in this way. Tensile breaking loads were determined on samples of two-ply yarn by a method described previously.[2] The standard of comparison for the strength of the coated samples was the strength of uncoated Thornel 50, two-ply yarn. This control value (Table I) is the average of 135 tensile tests on samples which were processed at various temperatures from 1023 K (750°C) to 1673 K (1400°C) without coating gases in the CVD coater. In all other respects, processing of the control samples was identical to that used in the regular coating runs.

The coat thickness vs strength behavior is graphically given in Fig. 2. Small amounts of TiC coat (~ 0.06 μm) appear to have significantly increased the strength of the Thornel 50 yarn, over that of the control. Above this it decreases rapidly, dropping below the control value at a thickness of about 0.12 μm, and reaching almost insignificant values above 0.15 μm. The rapid decrease in strength for TiC coat thicknesses above about 0.1 μm might be expected, because of the typical brittle, notch-sensitive nature of refractory metal carbides. For thinner coats, however, the brittleness of the TiC coat apparently has not yet become an important influence; its strength, however, seems to have contributed to the overall strength of the fibers.

The data of Fig. 2 show that there is a relatively narrow range of coat thicknesses over which TiC coated fibers can have useful strength. This strength limitation raises the question of how useful such thin coats can be as protective barriers. As will be shown below, very thin coats can provide significant protection.

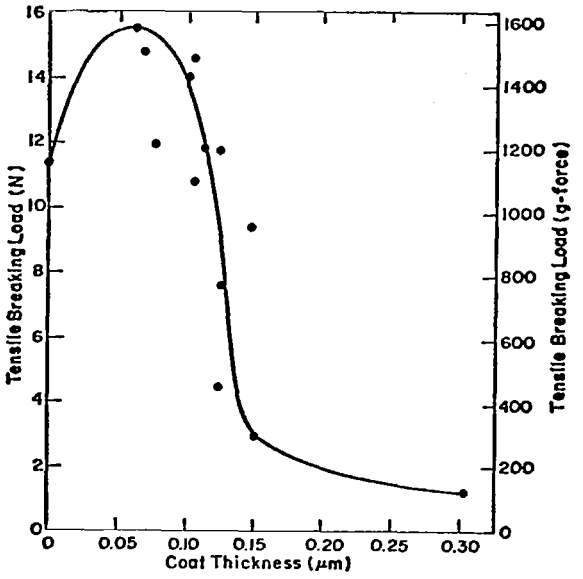


Fig. 2. Tensile breaking load vs coat thickness for TiC-coated Thornel 50 yarns.

SiC Coats.-$SiCl_4$ and CH_3SiCl_3 were used in a batch CVD apparatus to produce SiC coats on Thornel 50 fibers. The vapor coating apparatus, the processing procedures and the chemistry of the deposition process have been discussed previously.[1,3]

The following chemical reactions control the deposition of these gases and lead to formation of SiC coats on graphite substrates:

$$SiCl_4(g) + 2H_2(g) + C(s) = SiC(s) + 4HCl(g) \quad (1)$$

$$SiCl_4(g) + CH_4(g) = SiC(s) + 4HCl(g) \quad (2)$$

$$CH_3SiCl_3(g) = SiC + 3HCl \quad (3)$$

For the reaction of Equation 1, the carbon necessary for the formation of the metal-carbide is provided by the graphite fiber substrate. For the other reactions, the carbon is supplied in the coating gas, either as CH_4 (equation 2), which is added separately, or as part of the CH_3SiCl_3 molecule (equation 3). Besides the coating gases, helium is added as a diluent, together with excess hydrogen. The deposition kinetics can be varied by varying the deposition temperature and the proportions of the gases.

Twenty-nine CVD runs were made in which SiC coats were deposited on Thornel 50 fibers. Deposition conditions, tensile breaking loads, and microscopic appearances of the coated fiber yarns are listed in Table II. The breaking loads were determined by a method previously described.[2] None of the SiC coats was uniform or reproducible.

Some of the surface morphologies, typical of the coats produced, are shown in Fig. 3, together with the corresponding SEM x-ray scans. They vary from thin, incomplete coats (Fig. 3a) with almost undetectable Si counts by SEM x-ray scanning, to coats comprised of large nodular SiC crystals (Fig. 3b). More common, however, were thin, incomplete coats on which many nodules and tendrils had grown (Figs. 3c and 3d). There seemed to be no discernible difference between the coats produced from either of the coating gases, $SiCl_4$ or CH_3SiCl_3, used with or without CH_4. Furthermore, there were no obvious relationships between coat morphology, and coating time or temperature.

SiC coats on graphite fibers can significantly influence fiber strength. Tensile breaking loads of SiC-coated Thornel 50 yarn are plotted in Fig. 4 vs CVD coating temperature. Here, the coating gas was $SiCl_4$ and coating time was 15 min. Although there is considerable scatter in the data, the trend

TABLE II

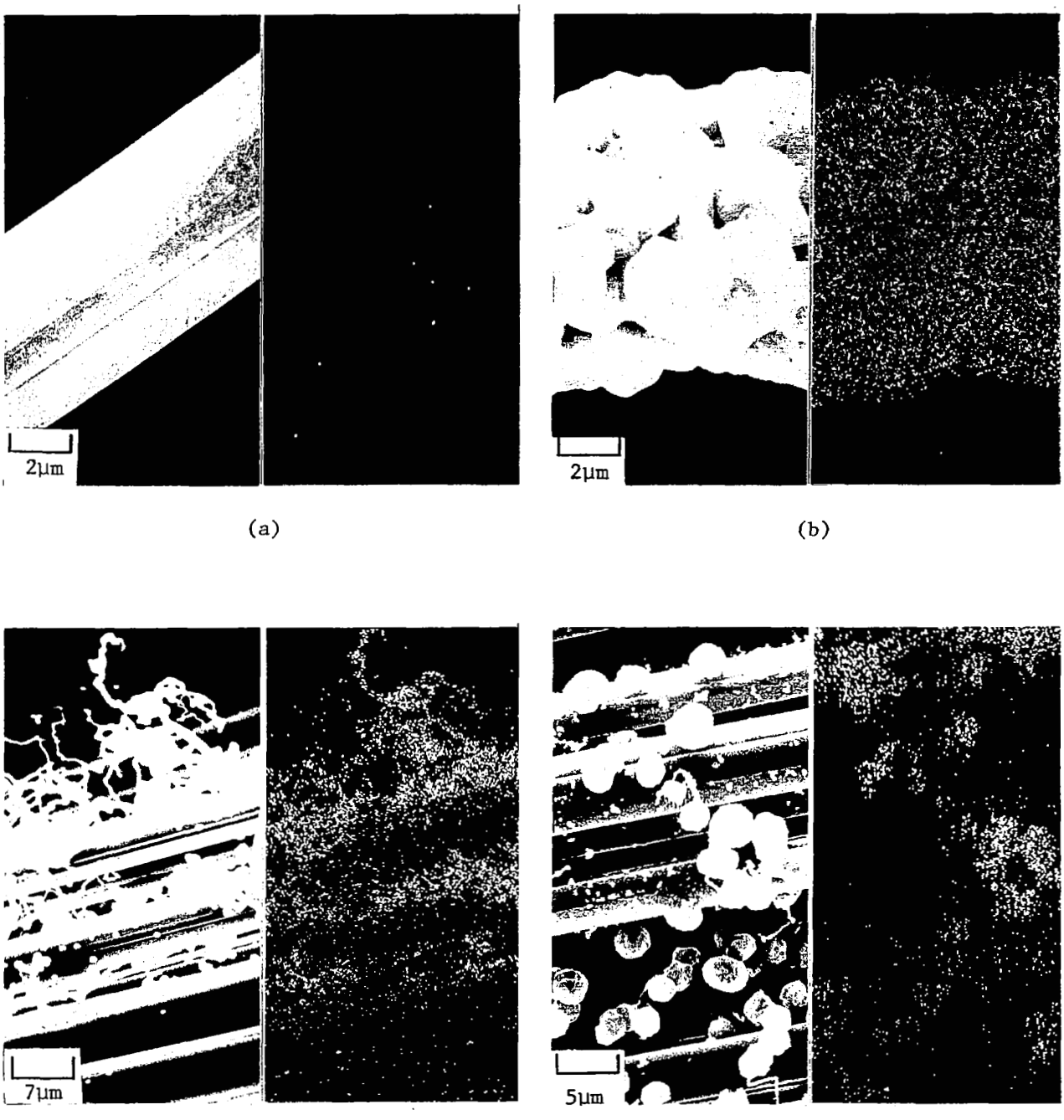
PROCESS CONDITIONS AND STRENGTHS FOR CARBON FIBER[a] SiC-COATING RUNS

Run No.	Deposition: Temp, K (°C)		Time, min	Coating Gas Flow Rate, STP, ℓ/min $SiCl_4$	CH_3SiCl_3	CH_4	H_2	HCl	He	SiC Coat Microscopic Appearance	Tensile Break Load, N (g-force) Mean		Std. Dev.	
8-29-74	1673	(1400)	120	0.28	0	0.1	5	1	3.0	thin, smooth, some nodules	7.94	(810)	1.30	(133)
8-30-74	1773	(1500)	120	0.34	0	0.1	5	1	3.5	large crystals, many welds	2.37	(242)	1.31	(134)
9- 3-74	1773	(1500)	15	0.18	0	0.1	5	1	3.5	v. thin, smooth, some nodules	10.32	(1052)	0.71	(72)
9- 4-74	1873	(1600)	15	0.58	0	0.1	5	1	3.5	v. thin, smooth, few clustered nodules	10.60	(1081)	1.73	(176)
9- 5-74	1573	(1300)	15	0.58	0	0.3	10	0	3.5	v. thin, many nodules and tendrils, welds	7.28	(742)	1.99	(203)
9- 6-74	1573	(1300)	15	0.55	0	0.1	10	0	3.5	v. thin, many nodules and tendrils, welds	9.80	(999)	1.54	(157)
9- 9-74	1573	(1300)	15	0.45	0	0	10	0	3.5	v. thin, some nodules and tendrils	12.34	(1258)	1.74	(177)
9-10-74	1473	(1200)	15	0.53	0	0	10	0	4.0	almost undetectable	11.31	(1153)	1.33	(136)
9-11-74	1473	(1200)	15	0.53	0	0	10	0	4.0	almost undetectable	11.49	(1172)	1.76	(179)
9-12-74	1423	(1150)	15	0.42	0	0	10	0	4.0	almost undetectable	14.16	(1444)	1.27	(130)
9-13-74	1373	(1100)	15	0.55	0	0	10	0	4.5	almost undetectable, some tendrils	12.71	(1296)	1.10	(112)
10-16-74[b]	1423	(1150)	15	0.47	0	0	10	0	4.5	varied: smooth to long "spikes", many welds	4.11	(419)	1.90	(194)
10-17-74	1423	(1150)	15	0.53	0	0	10	0	4.5	almost undetectable	10.47	(1068)	1.99	(203)
10-18-74[b]	1373	(1100)	15	0.53	0	0	10	0	4.5		4.07	(415)	1.02	(104)
11-13-74	1723	(1450)	15	0	0.36	0	2	0	2.0	"wormy" crust with many fine nodules	1.98	(202)	1.44	(147)
11-14-74	1723	(1450)	15	0	0.33	0	2	0	2.0	thick crystalline crust, inner fibers uncoated	4.41	(450)	2.00	(204)
11-15-74	1723	(1450)	15	0	0.24	0	2	0	2.0	varied: crusty coats of fine nodules to v. thin	6.29	(641)	1.60	(163)
11-18-74	1623	(1350)	15	0	0.27	0	2	0	2.0	varied: nodules, welds, tendrils, v. thin	13.85	(1412)	3.31	(338)
11-19-74	1723	(1450)	5	0	0.39	0	2	0	2.0	v. thin, nodules	10.30	(1050)	2.08	(212)
11-20-74	1823	(1550)	5	0	0.36	0	2	0	2.0	varied: many uncoated, numerous nodules on some	9.83	(1002)	1.45	(148)
11-21-74	1823	(1550)	15	0	0.27	0	2	0	2.5	varied: many uncoated, numerous nodules on some	10.51	(1072)	0.69	(70)
11-22-74	1823	(1550)	15	0	0.36	0	2	0	2.5	varied: many uncoated, numerous nodules on some	12.00	(1224)	1.67	(170)
11-26-74	1423	(1150)	15	0.47	0	0	10	0	4.0	thin, smooth, some welds	9.50	(969)	0.31	(32)
11-27-74	1423	(1150)	30	0.29	0	0	10	0	2.5	mostly uncoated with a few tendrils	13.47	(1374)	1.31	(134)
12- 2-74	1423	(1150)	60	0.31	0	0	10	0	4.0	mostly uncoated, tendrils and a few lg nodules	11.97	(1221)	3.01	(307)
12- 4-74	1423	(1150)	120	0.23	0	0	10	0	2.5	mostly uncoated, some large nodules and tendrils	9.71	(990)	0.46	(47)
12-16-74	1673	(1400)	15	0.45	0	0	10	0	2.5	many with thin coats, some uncoated, tendrils	8.67	(884)	0.82	(84)
12-17-74	1773	(1500)	15	0.40	0	0	10	0	2.5	numerous nodules, many welds	5.51	(562)	1.75	(178)
12-18-74	1873	(1600)	15	0.42	0	0	10	0	2.5	numerous large nodules, many welds	1.16	(119)	0.91	(93)
Control	—	--	--	0	0	0	10	0	5	uncoated	11.36	(1158)[c]	1.78	(182)

[a] Thornel 50, two-ply yarn.

[b] Diffuser baffle missing from coating crucible during run.

[c] Average of 135 tests on samples processed between 1023 K (750°C) and 1673 K (1400°C) without coating constituents in the gases.



(a) (b)

(c) (d)

Fig. 3. SEM photomicrographs of surface and Si x-ray images of Thornel 50 fibers CVD-coated with SiC in Runs: (a) 9-12-74, (b) 8-30-74, (c) 9-5-74 and (d) 11-18-74.

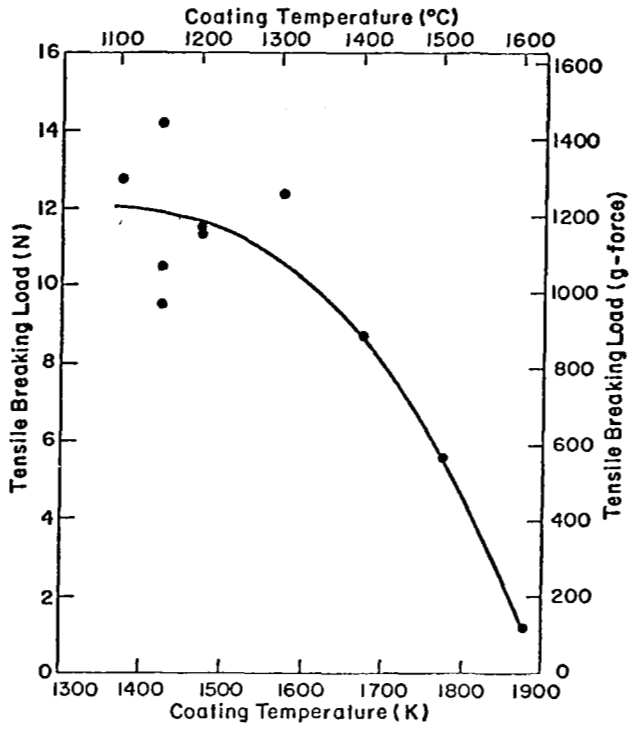


Fig. 4. Strength of SiC-coated Thornel 50 graphite yarn vs CVD coating temperature. Coating gas was $SiCl_4$. Coating time = 15 min.

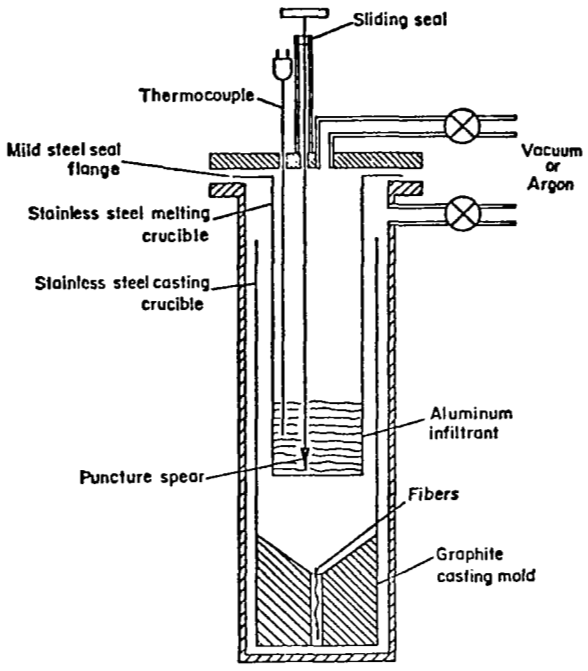


Fig. 5. Schematic of apparatus for pressure-infiltration of carbide-coated graphite fibers.

appears to be similar to that observed previously for TiC-coated fibers.[2] That is, strength decreases dramatically with increasing deposition temperature.

Infiltration

Successful infiltrations of carbide-coated carbon fibers have been made using an aluminum alloy. Samples were pressure-infiltrated in an apparatus based on a design by Banker,[4] shown schematically in Fig. 5. The infiltration procedure is as follows: The melting and casting crucibles are evacuated to a pressure of about 1 Pa ($\sim 10^{-2}$ torr). The vessel is placed into a vertical, resistance heated tube furnace, which is preheated to 943 K (670°C). When the infiltrant alloy has melted, the bottom of the melting crucible is punctured with the spear, allowing the alloy to flow into the casting mold containing the carbon fibers. The apparatus is then pressurized at 0.69 MPa (100 psi) with argon for approximately 8 min., removed from the furnace and allowed to cool under pressure to room temperature. Both TiC- and SiC-coated Thornel 50 yarns were infiltrated by this procedure.

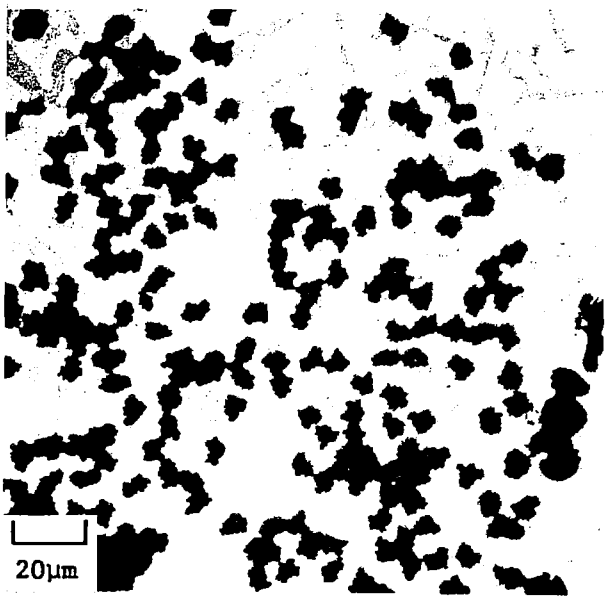
A typical example of the infiltration of TiC-coated Thornel 50 fibers is shown in the photomicrograph of Fig. 6a. Here, the coat thickness was approximately 0.1 μm (sample 8-14-75, Table I). For comparison, a sample of identically infiltrated uncoated Thornel 50 yarn is shown in Fig. 6b, and a commercially infiltrated material produced by Fiber Materials, Incorporated (FMI) is shown in Fig. 6c. The FMI product contains Thornel 50 yarn which was chemically treated by a proprietary process to promote wetting. The infiltrant for the two LASL-produced materials (Figs. 6a and 6b) was the Al-13% Si alloy discussed above. That used for the commercial material (Fig. 6c) was reported by FMI also to be Al-13% Si. As seen, infiltration of the TiC-coated fibers was essentially as good as that of the commercially treated yarn. The uncoated fibers, however, underwent essentially no infiltration or wetting. A series of infiltrations was made on a group of TiC-coated Thornel 50 yarns which had coat thicknesses which varied from 0.06 μm to 0.30 μm. All samples appeared to infiltrate equally well.

In Fig. 7, four examples of infiltrated, SiC-coated Thornel 50 fibers are given. The fibers used for these infiltrations are those shown in Fig. 3, and represent typical examples of the SiC-coating series (Table II). Here, as above, the infiltrant was the LASL Al-13% Si alloy. Figures 7a and 7b show the extremes in coat thickness for this series. At one extreme (Fig. 7a) is the poorly coated yarn from CVD run 9-12-74 which had very little detectable SiC coat on the individual fibers, and which underwent essentially no infiltration or wetting by the aluminum alloy. The fibers used for the infiltration of the sample shown in Fig. 7b, however, had a thick coat composed, mainly, of large SiC crystals, and underwent extensive infiltration. The fibers of the two samples shown in Figs. 7c and 7d were typical of most produced in the SiC coating series and, in general, had thin, incomplete coats with many nodules and tendrils. For these, infiltration was only moderate, at best.

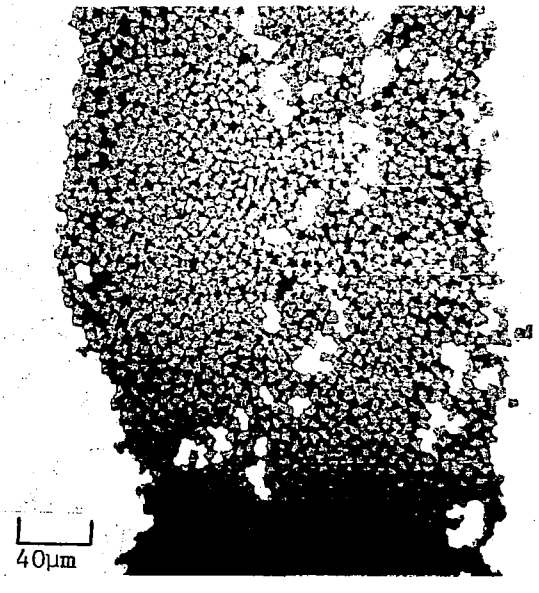
Diffusion Barrier Experiments

To evaluate the effectiveness of thin carbide coats as diffusion barriers to prevent reaction between carbon and aluminum, samples of Al-infiltrated Thornel 50 fibers, coated with either TiC or SiC, were heated for prolonged periods at elevated temperatures. Sample compositions, heat-treatment temperatures, infiltrant solidus temperatures and microscopic appearances of the treated samples are summarized in Table III. For comparison, the table also includes data for similarly-treated samples (AD-10A, -10B and -10C) of commercially treated Thornel 50 yarns, infiltrated by FMI. All samples were heated in purified argon for 100 hours at temperatures ranging from 753 K (480°C) to 953 K(680°C). Solidus temperatures of the LASL infiltrants correspond to the thermal arrest temperatures observed, on heating, during infiltration of the carbide-coated fibers. For the FMI material the solidus temperature used was that for the standard A13 casting alloy.[5] The fibers in samples AD-11A, -11B, and -11C had TiC coats approximately 1/8 μm thick. The SiC-coated fibers in samples AD-12A and -12B, however showed barely detectable amounts of Si on their surfaces by SEM x-ray analysis.

At heat-treatment temperatures up to within 40 or 50 degrees of the infiltrant alloy solidus, no reaction was observed for any of the materials



(a)



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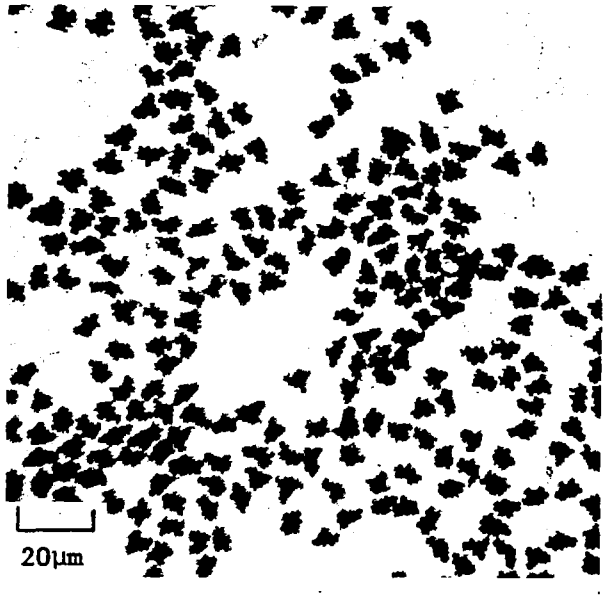


(c)

Fig. 6. Optical photomicrographs of Thornel 50 yarns infiltrated with Al-13% Si alloy. (a) TiC-coated fibers, (b) uncoated fibers, (c) commercially treated fibers (FMI).

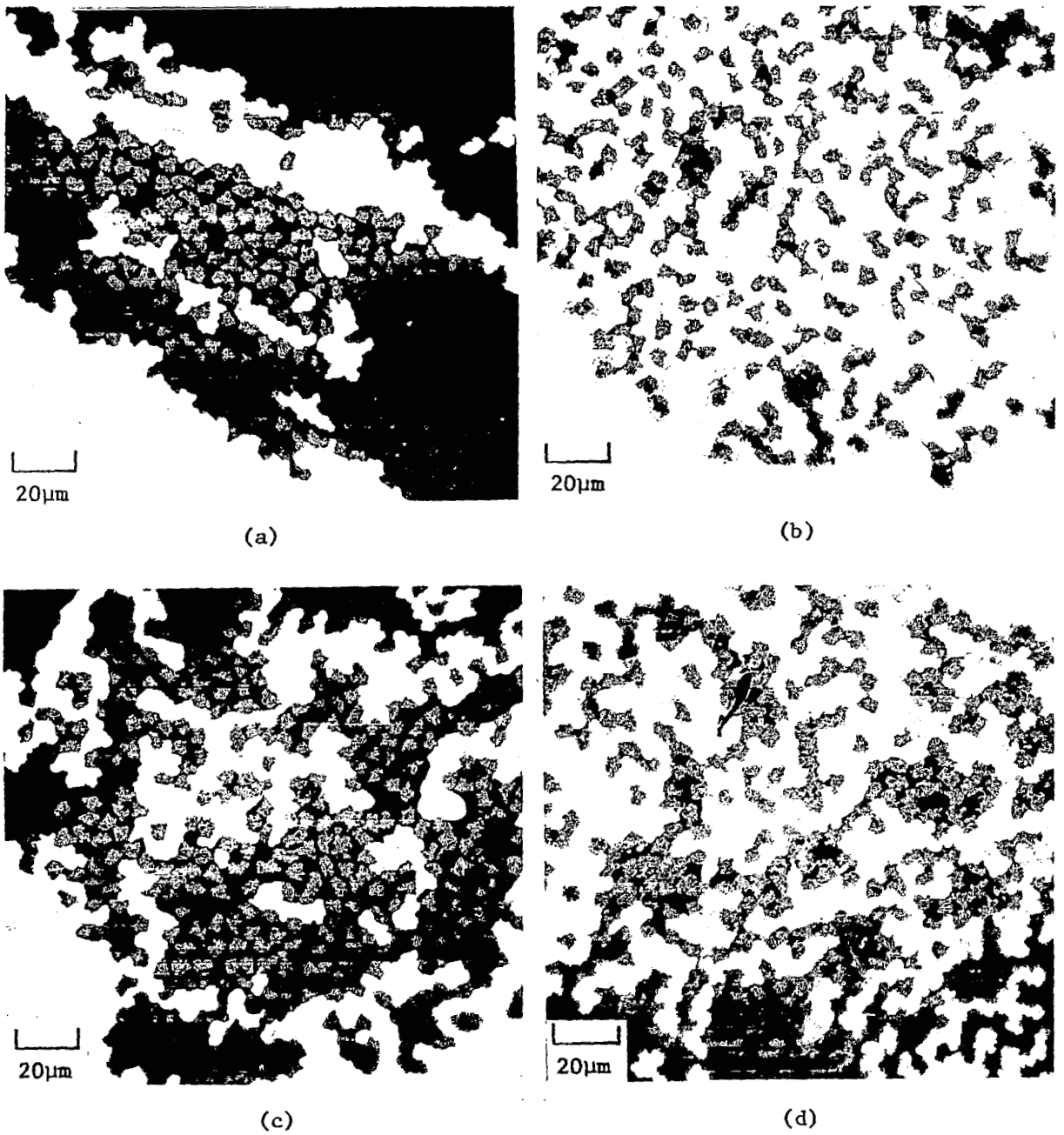


Fig. 7. Optical photomicrographs of SiC-coated Thornel 50 yarns infiltrated with Al-13% Si alloy. The coated fibers here correspond to those shown in Fig. 3 and Table II, as follows: (a) Run 9-12-74, (b) Run 8-30-74, (c) Run 9-5-74 and (d) Run 11-18-74.

TABLE III

DIFFUSION BARRIER EXPERIMENTS

Sample No.	Sample Composition (Alloy/Substrate)	Alloy Solidus K (°C)	Heat Treatment[a] K (°C)	Microscopic Appearance
AD-10A	Al-13% Si/Thornel 50 (FMI)[b]	847 (574)	753 (480)	no reaction
AD-10B	"	"	803 (530)	no reaction
AD-10C	"	"	953 (680)	extensive reaction
AD-11A	Al-13% Si/TiC-coated Thornel 50	843 (570)	753 (480)	no reaction
AD-11B	"	"	803 (530)	no reaction
AD-11C	"	"	953 (680)	reaction
AD-12A	Al-13% Si/SiC-coated Thornel 50	858 (585)	753 (480)	no reaction
AD-12B	"	"	953 (680)	extensive reaction

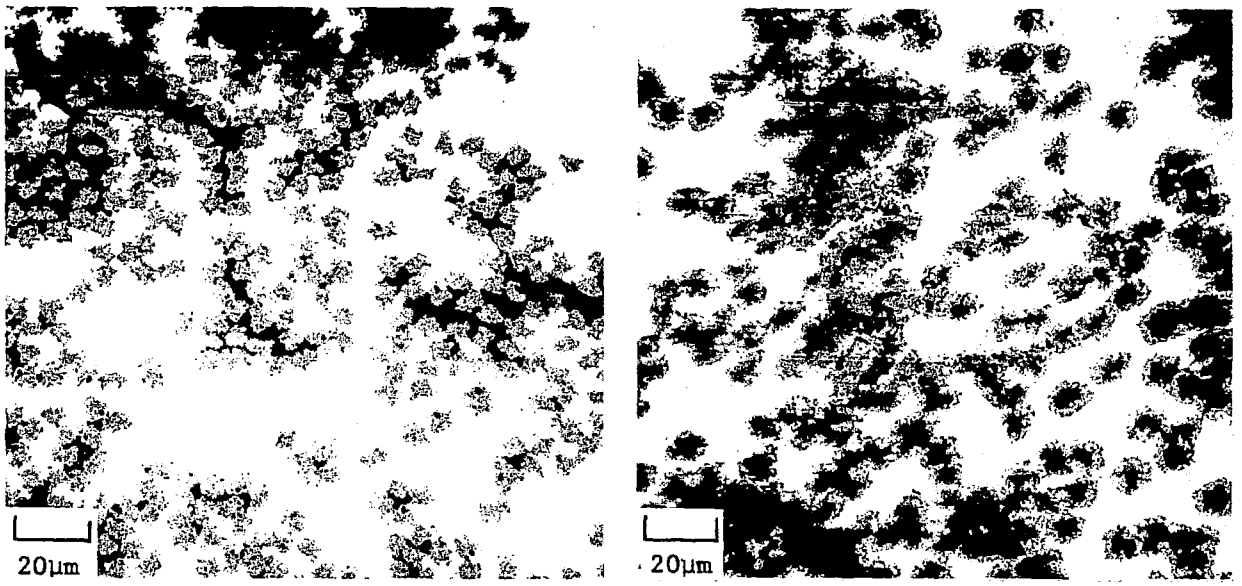
[a]Samples held at temperature for 100-hours.

[b]Thornel 50 fiber yarn infiltrated with Al-13% Si by Fiber Materials, Inc.

(Table III). Above the solidus all samples showed some reaction, particularly the commercially treated fibers which experienced extensive reaction. The very thin, nonuniform coats of SiC similarly provided little protection to the fiber substrate. Although the TiC-coated fibers also reacted with the aluminum alloy matrix above the solidus temperature, the reaction was not nearly so severe as it was for the commercially treated fibers or for those with the thin SiC coats. This is illustrated in Figs. 8 and 9 which show optical photomicrographs of infiltrated, TiC-coated fibers and commercially treated fibers, respectively, which were heated at temperatures above and below the solidus. The microstructures of the infiltrated SiC-coated fibers showed, essentially, an identical extent of reaction as shown for the FMI material in Fig. 9. Apparently, carbide coats with thicknesses as little as 1/8 μm can provide enough surface protection to inhibit reaction between graphite fibers and an aluminum alloy matrix at elevated temperatures.

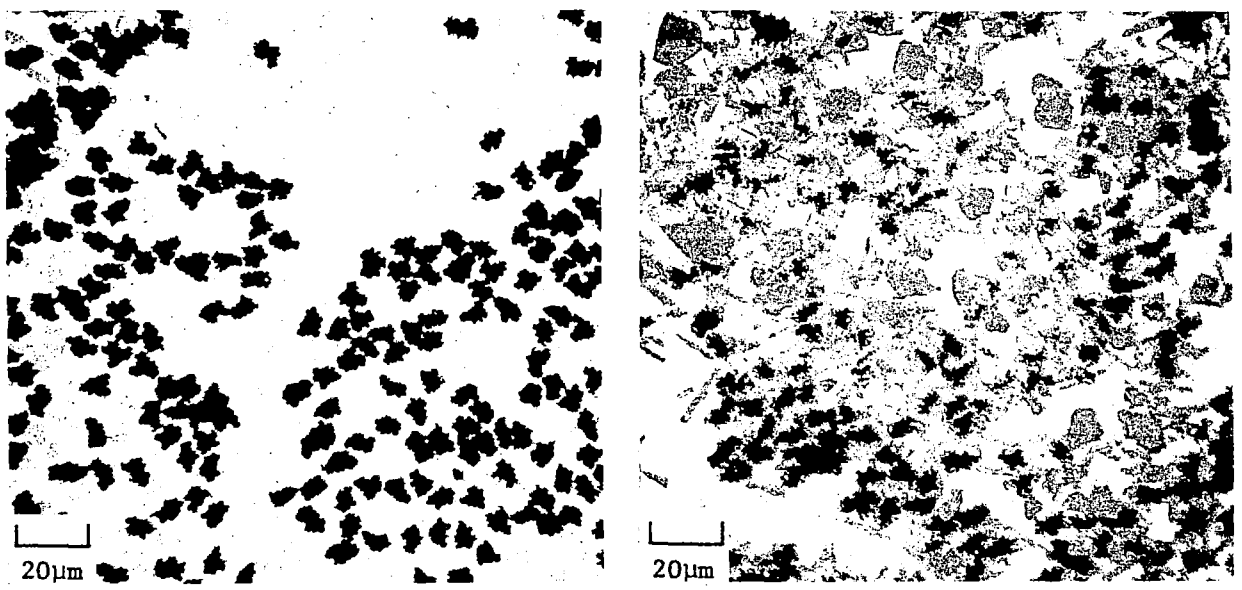
Compaction Experiments

Two consolidation runs were made to demonstrate the ability to compact Al-infiltrated graphite yarns into graphite-aluminum composite bars. These were made in the apparatus shown schematically in Fig. 10. In the first run, samples of Thornel 50 yarn, infiltrated by FMI with an Al-13% Si alloy, were cut into 12.7-mm (0.5-in.) lengths, placed in the graphite mold and compacted at 0.69 MPa (100 psi) and 903 K (630°C) in the vacuum chamber. The hot-pressed composite was 0.64-mm (0.025 in.) thick, 6.35-mm (0.25 in.) wide and 12.7-mm (0.5-in.) long. In the second run, samples of TiC-coated Thornel 50, also infiltrated with Al-13% Si alloy, were similarly compacted to a final thickness of 1.27-mm (0.050-in.). Photographs of the samples showing their appearances



(a) (b)

Fig. 8. Optical photomicrographs of TiC-coated Thornel 50 fibers, infiltrated with Al-13% Si alloy, then heated 100 hours in argon at (a) 753 K (480°C) (sample AD-11A) and (b) 953 K (680°C) (sample AD-11C).



(a) (b)

Fig. 9. Optical photomicrographs of commercially treated Thornel 50 fibers, infiltrated with Al-13% Si alloy, then heated 100 hours in argon at (a) 753 K (480°C) (sample AD-10A) and (b) 953 K (680°C) (sample AD-10C).

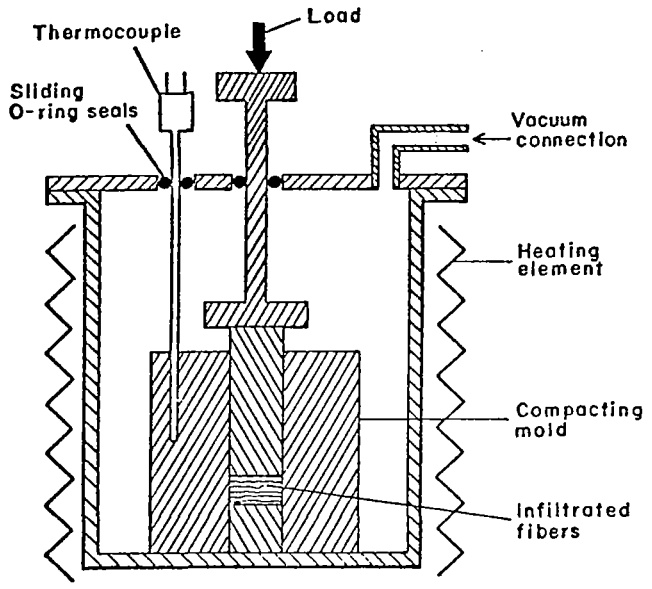


Fig. 10. Schematic of apparatus for forming composite bars from aluminum-infiltrated carbide-coated fibers.

after removal from the mold are given in Fig. 11, together with photomicrographs of their cross sections. The commercially treated fibers were compacted to a greater fiber concentration than were the TiC-coated fibers. This is probably due to a greater excess of alloy infiltrant in the coated fibers than in those commercially treated, and should be correctable by compacting at higher pressure to squeeze out excess infiltrant.

CONCLUSIONS

1. Thin coats of CVD-deposited TiC on graphite fibers were shown to enhance fiber strength, promote infiltration of the fibers with aluminum, and inhibit reaction between the graphite fibers and the aluminum at elevated temperatures.

2. SiC coats, produced by CVD on graphite fibers, were neither uniform nor controllable. They tended to be characterized by nodules and tendrils, but a few were so thin as to be nearly undetectable. Despite the nonuniformity of these coats, even the thin ones were able to promote some infiltration with aluminum.

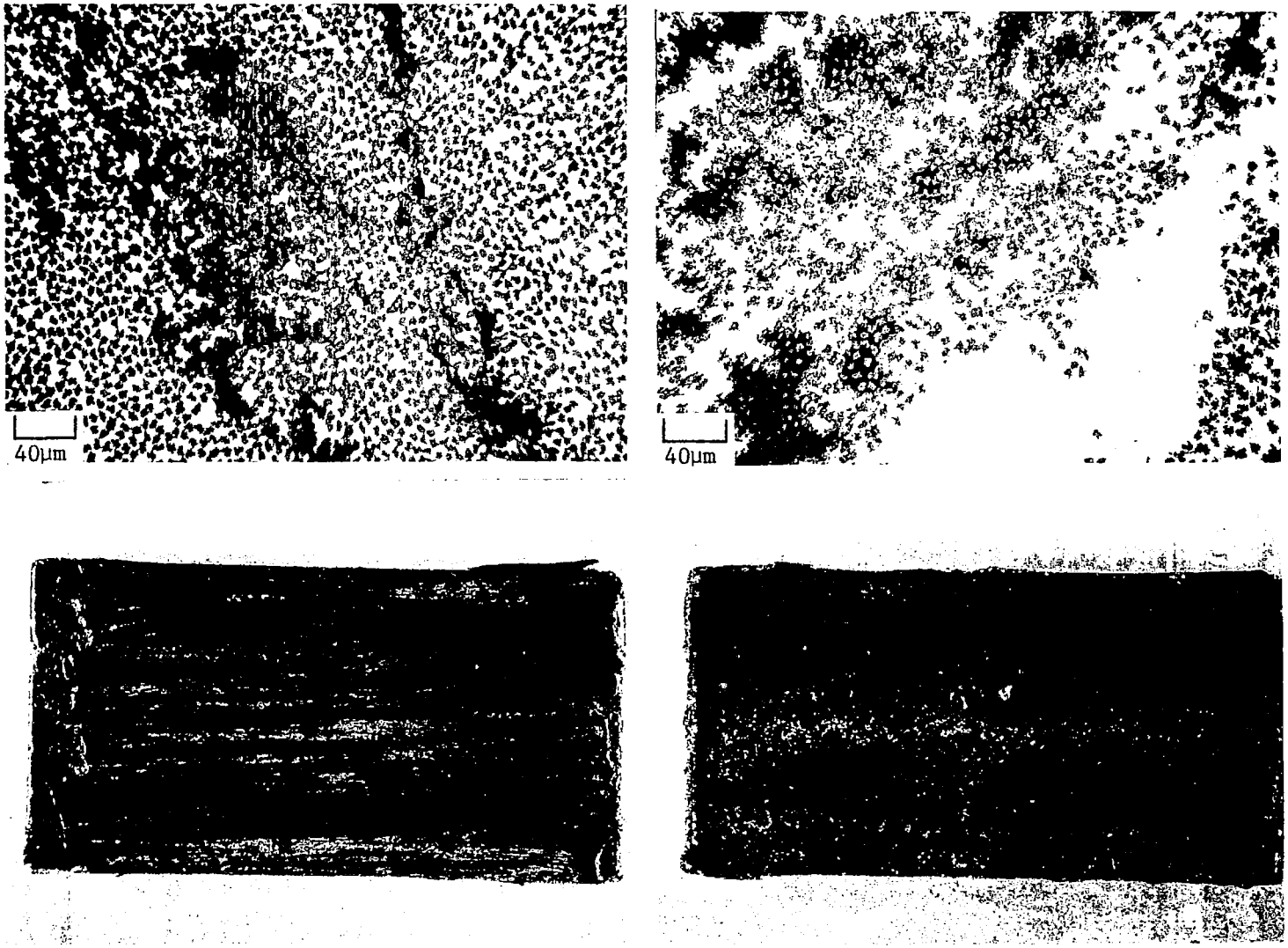
3. Small graphite-aluminum composite samples have been made from Al-infiltrated graphite yarn by vacuum hot-pressing at temperatures above the metal liquidus.

REFERENCES

1. Imprescia, R. J., Levinson, L. S., Reiswig, R. D., Wallace, T. C., and Williams, J. M., "Carbide Coated Fibers in Graphite-Aluminum Composites, Progress Report No. 1, September 1, 1973 - January 31, 1974," NASA CR-2533, 1975.

2. Imprescia, R. J., Levinson, L. S., Reiswig, R. D., Wallace, T. C., and Williams, J. M., "Carbide Coated Fibers in Graphite-Aluminum Composites, Progress Report No. 2, February 1 - July 31, 1974," NASA CR-2566, 1975.

3. Wallace, T.C., "Chemical Vapor Deporition of ZrC in Small Bore Carbon-Composite Tubes," Proceedings of the Fourth International Conference on Chemical Vapor Deposition, Boston, Massachusetts, October 7-11, 1973 pp. 91-106.



COMMERCIALLY TREATED FIBERS

TiC-COATED FIBERS

Fig. 11. Al-infiltrated Thornel 50 fibers compacted at 0.69 MPa (100 psi) and 903 K (630°C).

4. Banker, J. G., "Metal Matrix Composite Fabrication by Liquid Infiltration," SAMPE Quarterly, January 1974, p. 39.

5. Metals Handbook, 8th edition, Vol. 1 American Society for Metals, 1961, p. 950.